

No.	DGEBPA	Acrylic Acid	Diethanolamine	Water	Acrylic Acid	Visc.
1	36g (0.1 mol)	5.8g (0.08 mole)	12.6g (0.12 mol)	20g	10g	>1p
2	36g (0.1 mol)	5.6g (0.09 mol)	11.5g (0.11mol)	20g	7g	>1p
3	36g (0.1 mol)	7.2g (0.10 mol)	10.5g (0.10 mol)	20g	6g	420cp
4	36g (0.1 mol)	7.9g (0.11 mol)	9.5g (0.09 mol)	20g	6g	980cp
5	36g (0.1 mol)	8.6g (0.12 mol)	8.4g (0.08 mol)	20g	5g	110cp
6	36g (0.1 mol)	9.4g (0.13 mol)	7.4g (0.07 mol)	20g	6g	620cp
7	36g (0.1 mol)	10.1g (0.14 mol)	6.3g (0.06 mol)	20g	4g	>1p

Each sample was then coated onto calendared paper at 5 microns using a wire wound applicator, and passed under UV light at 15m/minute. Each film was examined for gloss, surface tack, hardness, water and MEK resistance. The results were as follows:

Examples 3, 4 and 5 all cured after one pass to give satisfactory films.

Examples 1 and 2 were more difficult to apply due their viscosity and required two passes to cure.

Examples 6 and 7 were slightly more turbid and produced dull films.

#### Example 8

This example and examples 9 to 21 below demonstrate the preparation of an epoxy amine resin which is reacted with an unsaturated acid to form a resin salt in accordance with the invention. One mole of bisphenol A diepoxide resin was reacted with at least 1 mole of diethanolamine to form an adduct. Allowing the exotherm of this reaction to take its course over 5-10 minutes. The composition was diluted with water or quenched into water to form a stable dispersion of the adduct. To this dispersion, 2 moles of acrylic acid or methacrylic acid were added to produce a cationic resin solution, with very similar properties of those resins formed from epoxy acrylate half ester described in Examples 1 to 7. These resins have low odour and colour and will photopolymerise, when exposed to UV light, to a hard tack-free state.

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The resin is of low viscosity and permits solids contents of at least 80%, with as little as 20% water, and have moderately low viscosity. The overall reaction time can be accomplished within 1 hour, by avoiding the longer ester formulation of the method described in Examples 1 to 7. The resins of this example appears to show reduced tendency to auto-polymerise.

### Example 9

Bisphenol di-glycidyl ether resin (1 mole) 360g is mixed with diethanolamine (1 mole) 105g at room temperature, and heated with stirring to 70°C, when it forms a clear homogeneous mixture. At this stage an energetic reaction takes place and the external heat is removed. The resultant exotherm increases the temperature rapidly to 140-150°C with a corresponding reduction in viscosity of the clear fluid. This is rapidly poured into (10 moles) 180g of cold water, with fast stirring to produce a white dispersion, with corresponding increase in temperature of the resin mixture. Acrylic acid (2 moles) 144g, is added over several minutes and a clear resin solution results. The resin solution is allowed to cool, with continuous stirring. The resultant resin is a clear, colourless, low viscosity oligomer solution with almost no odour and a pH of 4-6. When cast as a thin film onto a metal or paper substrate, and exposed to UV light for a few seconds, the resin rapidly produces a hard, tack-free and odour-free clear glossy film, with good water and solvent resistance.

### Examples 10-15

The procedure of Example 8 was followed with the exception that water was added to the resin at the stage of exotherm in which the resultant reflux of water assisted in controlling the heat and provided the dispersion prior to salt formation. Different amines and unsaturated acids were also examined.

Examples	Ex10	Ex11	Ex12	Ex13	Ex14	Ex15
	Moles	Moles	Moles	Moles	Moles	Moles
BPDGE (Araldite 6010)	1.0	1.0	1.0	1.0	1.0	1.0
Diethanolamine	1.0	1.0	1.3	1.6	1.0	1.0
Water	10.0	10.0	10.0	10.0	10.0	10.0
Acrylic acid	1.0	2.0	2.0	2.0	-	-
Methacrylic acid	-	-	-	-	1.0	2.0

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Each of these resin solutions was a clear low viscosity solution and produced a hard glossy film after exposure to UV radiation. Example 13 did show a tendency to water sensitivity at the higher ratio of diethanolamine.

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### Examples 16-21

Examples	Ex16	Ex17	Ex18	Ex19	Ex20	Ex21
	Moles	Moles	Moles	Moles	Moles	Moles
BPDGE (Araldite 6010)	1.0	1.0	1.0	1.0	1.0	1.0
Diethanolamine	1.0	1.0	1.3	1.6	1.0	1.0
Diethylamine	-	1.0	0.7	0.4	-	-
Urea	-	-	-	-	0.5	1.0
Water	10	10	10	10	10	10
Acrylic acid	2.0	2.0	2.0	2.0	2.0	2.0

These resins displayed differences in viscosity, but each was photocurable and produced satisfactory films.

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### Examples 22-28

These examples examine preparation of compositions of this invention using melamine formaldehyde based resins.

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#### Example 22

To 324g of 37% formalin (4 moles) is added 126g of melamine (1 mole) at room temperature and heated with stirring, to 70°C and held until clear, about 40 minutes. The solution is cooled to 45°C and 145g of 40% glyoxal (1 mole) is added with constant stirring, and held for a further 30 minutes. A clear pale amber solution results, to which is added 144g of acrylic acid (2 moles), and allowed to cool. The resultant solution is a very pale, clear and odour-free solution which when cast onto a metal or paper substrate and exposed to UV radiation, will cure rapidly to a hard, tack-free and odour-free clear film, with good water and solvent resistance.

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The procedure of Example 22 was repeated using various proportions of components and alcohols as shown in the table below.

Examples	Ex23	Ex24	Ex25	Ex26	Ex27	Ex28
	Moles	Moles	Moles	Moles	Moles	Moles
Formalin 37%	3	3	3	3	3	3
Glyoxan 40%	1	1	1	1	1	1
Melamine	1	1	1	1	1	1
Ethanol	-	2	-	-	2	-
n-Propanol	-	-	2	-	-	2
Hydroxy ethyl acrylate	-	-	-	1	-	-
Acrylic acid	2	2	2	-	1	2

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**Example 29**

In a similar manner, to 324g of 37% formalin (4 moles) is added 126g of melamine and also 240g of n-propanol (4 moles) and heated with stirring to 65°C and held for 30 minutes. A clear solution results which smells faintly only of propanol, to which is added 144g of acrylic acid (2 moles), and the stirring continued for a further 30 minutes at 50°C, then cooled. This resin solution behaves in the same way as Example 12, will UV cure to a clear glossy film with faint odour of propanol. It does appear to be slightly more flexible in thicker films than Example 22 films.

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**Example 30-33**

Resins were prepared according to Example 29 but replacing n-propanol with methanol (Example 30), ethanol (Example 31), butanol (Example 32) and isopropanol (Example 33). Each resin responded to UV radiation. The isopropanol resin was the least reactive thought to be due to steric hindrance. The resin based on n-butanol had a stronger residual odour.

**Example 34**

stirring. 1 mole (72g) of acrylic acid was slowly added. On testing for cure, this resin was found to cure only with 1-2% of photoinitiator (Irgacure 184).

**Example 41 and Comparative Example A**

- The composition of Example 20 was formed into a coating composition for use  
5 as a high gloss overprint varnish having the following compositions

**Example 41**

Component	Parts by Weight
Epoxy acrylate (Ex 20)	80.0
PA57 silicone glycol copolymer levelling agent	0.5
"Quatramine C16/50"	4.5
(50% active solution of trimethyl hexadecyl ammonium chloride in a mixture of water and ethanol (levelling agent))	
Water	15.0
	100.0

- 10 Viscosity Brookfield RVT #4/100/25°C = 460 cps.

- The composition was drawn down to a 5 micron coating on printed paper stock and cured using a mercury UV lamp of 80 w/cm at half power (ie 40 w/cm) half  
15 speed of 35 meters per min to provide a UV lamp energy of 25 millijoules per cm<sup>2</sup>. The radiometer used to measure energy was a EIT Uvicure Radiometer model M365. The composition of the invention cured without addition of photoinitiator.

- 20 A conventional UV curable high gloss overprint varnish was prepared with the following composition.

**Comparative Example A**

<b>Component</b>	<b>Parts by Weight</b>
Craynor CN104 A80 = Epoxy Acrylate cut with 20% T.P.G.D.A.	43.0
T.M.P.E.O.T.A. = Ethoxylated Trimethylolpropane Triacrylate	48.5
Methyldiethanolamine = Amine synergist	3.0
Benzophenone, Photoinitiator	3.0
Irgacure 184 = 1-Hydroxy-cyclohexyl-phenyl-ketone, Photoinitiator	2.0
PA57 = Silicone glycol Copolymer (levelling and slip agent)	0.5
	100.0

- 5 Viscosity = Brookfield RVT #4/100/25°C = 460 cps

The prior art composition required 5% photoinitiator and 3% amine synergist to cure under the same conditions.

- 10 Those skilled in the art will appreciate that there may be many variations and modifications of the configuration described herein which are within the scope of the present invention.

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Ms Susan W Berman  
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US Patent and Trademark Office


Dear Ms Berman

Re: Robert John Bolton  
United States Application No. 09/936,599  
Title: Radiation Curable Resin Composition  
Our Reference: FF34319/01

As requested we are forwarding herewith the replacement pages 12 to 15 and 18 to 19 for this application minus the grey shading in the tables for easier readability and printing.

We apologise for the inconvenience and thank you for prompt assistance.

Yours sincerely  
PHILLIPS ORMONDE & FITZPATRICK



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